

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

HAYNS=1

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

09/202500INTERNATIONAL APPLICATION NO.
PCT/GB97/01319INTERNATIONAL FILING DATE
14 May 1997PRIORITY DATE CLAIMED
16 May 1996

TITLE OF INVENTION
LIQUID AND GAS PURIFICATION AND FILTRATION

APPLICANT(S) FOR DO/EO/US
PCT/GB97/01319

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
- ☒ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:
 1. A courtesy copy of the specification as originally filed.
 2. A courtesy copy of the first page of the International Publication (WO97/43028).
 3. A courtesy copy of the International Search Report.
 4. A courtesy copy of the International Preliminary Examination Report with annexes.
Note: Please use pages 1-3 of the specification and the claims as they appear in the IPER annexes in place of originally filed pages 1-3 of the specification and originally filed claims for examination in this case.
 5. Formal drawings, 9 sheets, figures 1-10.

17. ☒ The following fees are submitted:**BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :**

Neither international preliminary examination fee (37 CFR 1.482)
nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO
and International Search Report not prepared by the EPO or JPO \$970.00

International preliminary examination fee (37 CFR 1.482) not paid to
USPTO but International Search Report prepared by the EPO or JPO \$840.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but
international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$760.00

International preliminary examination fee paid to USPTO (37 CFR 1.482)
but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$670.00

International preliminary examination fee paid to USPTO (37 CFR 1.482)
and all claims satisfied provisions of PCT Article 33(1)-(4) \$96.00

ENTER APPROPRIATE BASIC FEE AMOUNT =**CALCULATIONS** PTO USE ONLY

\$ 840.00

\$ 130.00

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☒ 30
months from the earliest claimed priority date (37 CFR 1.492(e)).

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE
Total claims	17 - 20 =		X \$18.00
Independent claims	3 - 3 =		X \$78.00
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$260.00

\$ 970.00

TOTAL OF ABOVE CALCULATIONS =

Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement
must also be filed (Note 37 CFR 1.9, 1.27, 1.28).

\$

SUBTOTAL =

\$ 970.00

Processing fee of \$130.00 for furnishing the English translation later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(f)).

\$

TOTAL NATIONAL FEE =

\$ 970.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +

\$

TOTAL FEES ENCLOSED =

\$ 970.00

Amount to be:
refunded \$
charged \$

a. ☒ A check in the amount of \$ 970.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees.
A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any
overpayment to Deposit Account No. 02-4035. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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SIGNATURE:

Roger L. Browdy

NAME

25,618

REGISTRATION NUMBER

Date of this submission: December 16, 1998

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	Art Unit:
Andrew B. HAYNS)	
IA No.: PCT/GB97/01319)	
IA Filed: 14 May 1997)	Washington, D.C.
U.S. App. No.:)	
(Not Yet Assigned))	
National Filing Date:)	December 16, 1998
(Not Yet Received))	
For: LIQUID AND GAS...)	Docket No.: HAYNS=1

PRELIMINARY AMENDMENT

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Contemporaneous with the filing of this case and prior
to calculation of the filing fee, kindly amend as follows:

IN THE CLAIMS

Claim 3, line 1, delete "or 2".

Claim 4, lines 1 and 2, delete "2 or 3,".

Claim 5, lines 1 and 2, delete "2 or 3,".

Claim 6, lines 1 and 2, delete "2 or 3,".

Claim 7, lines 3 and 4, delete "any of claims 1 to 6",
and insert therefor --claim 1--.

Claim 8, line 3, delete "any one of claims 1 to 6", and
insert therefor --claim 1--.

Claim 12, line 1, delete "or 11".

Claim 13, lines 1 and 2, delete "any one of claims 10 to
12", and insert therefor --claim 10--.

In re of HAYNS=1

Claim 17, lines 3 and 4, delete "any one of claims 1 to 6", and insert therefor --claim 1--.

REMARKS

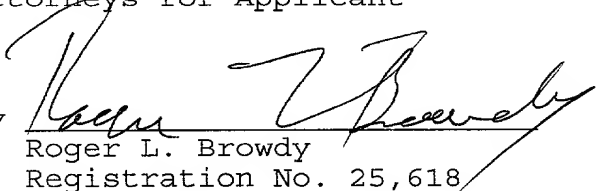
The above amendments to the claims are being made in order to eliminate any properly multiply dependent claims, for the purpose of reducing the filing fee. Please enter this amendment prior to calculation of the filing fee in this case.

Favorable consideration and allowance are earnestly solicited.

Respectfully submitted,

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	Art Unit:
Andrew B. HAYNS)	
IA No.: PCT/GB97/01319)	
IA Filed: 14 May 1997)	Washington, D.C.
U.S. App. No.:)	
(Not Yet Assigned))	December 16, 1998
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(Not Yet Received))	
For: LIQUID AND GAS...)	Docket No.: HAYNS=1

SUPPLEMENTAL PRELIMINARY AMENDMENT

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Prior to examination on the merits, kindly amend as follows:

IN THE CLAIMS

Please amend the claims as follows:

Claim 11, line 1, replace "12" with --10--.

REMARKS

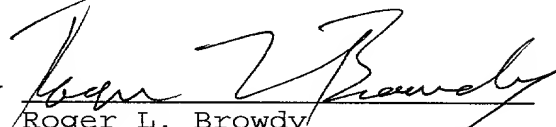
Claims 1-17 presently appear in this case. The present amendment is being made to correct a typographical error and to place this case into better condition for examination.

In re of HAYNS=1

Favorable consideration and allowance are earnestly
solicited.

Respectfully submitted,

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09/202500

LIQUID AND GAS PURIFICATION AND FILTRATION

5 The present invention relates to the removal of
organic and other pollutants from liquids and gases,
and in particular, but not exclusively, to the removal
of such pollutants by a filtration system.

10 A number of strategies have been developed in the
petrochemical industry for dealing with problems such
as oil spillage and leakage, particularly at sea. Some
methods, for example the use of detergents, simply aim
to disperse the oil spillage as quickly as possible
before too much damage has been done. It is, however,
15 preferable to remove the oil from the water without
allowing it to disperse, since there are many toxic
components in the oil which may cause harm to the
environment. It is known to provide a granular
material based on cellulose, which has oil-absorbing
properties, the material being in a form suitable for
20 sprinkling onto an oil spillage. Once the oil has been
absorbed, the material is gathered up and may be
incinerated.

25 Oil spillages are not the only environmental
problem faced by the petrochemical industry. There are
many situations where it is desirable to remove
components including organic pollutants (such as
hydrocarbons) and heavy metal contaminants from
produced water and water run-off before this water is
released as effluent.

30 It is also desirable to remove such pollutants
from liquids other than water and also from gases (e.g.
air).

35 According to a first aspect of the present
invention, there is provided a filter material
comprising a matrix in which is dispersed a granular
formulation of a material comprising a base formed

substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms.

5 In preferred embodiments, the hydrocarbon chains of the one or more carboxylic acids consist of 10 to 18 carbon atoms. Particularly effective carboxylic acids have been found to include stearic acid $\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$ and palmitic acid $\text{CH}_3(\text{CH}_2)_{14}\text{COOH}$.

10 According to a second aspect of the present invention, there is provided a method of producing a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids
15 having hydrocarbon chains consisting of 8 to 20 carbon atoms, wherein one or more cellulosic materials are mixed together with a powdered formulation of the one or more carboxylic acids.

20 The granular material of the first aspect of the present invention may be formed by mixing together one or more cellulosic materials, for example virgin pulp and wood chips, together with the one or more carboxylic acids in powder form and, optionally, latex. The mixing is preferably undertaken in a hammer mill,
25 in which heat and friction assist the process whereby the carboxylic acid becomes adsorbed onto the cellulose fibres. It is thought that the carboxylic acids are adsorbed onto the surface of the cellulose fibres by way of the carboxyl $-\text{COOH}$ functional group, either
30 through hydrogen bonding or through the formation of cellulose esters containing an $-\text{O}-\text{CO}-\text{R}$ group formed with the hydroxyl $-\text{OH}$ groups on the cellulose rings. However the carboxylic acids are bonded to the cellulose fibres, the result is that the material of
35 the first aspect of the present invention comprises cellulose fibres from which project hydrophobic

hydrocarbon chains. When the material is applied to a mixture of water and hydrocarbon pollutants, the hydrophobic hydrocarbon tails of the carboxylic acid residues serve to attract the hydrocarbon pollutants to the material and to repel water, thereby providing the required separation. The material, incorporating the hydrocarbon pollutants, can then be gathered up and used as a fuelstock.

The matrix of the first aspect of the invention may be fabricated from a number of materials, including non-woven fibrous materials, open-cell foam materials or a cotton or viscose gauze. The unloaded matrix advantageously has a density not greater than 0.25gcm^{-3} , and preferably from 0.01 to 0.18gcm^{-3} . A particularly preferred matrix has a thickness of around 3mm and a density in the region of 0.1gcm^{-3} . The granular formulation of the material of the first aspect of the present invention may be incorporated into the matrix by bombardment across a pressure gradient as described in EP 0 504 214, the disclosure of which is hereby incorporated by reference into the present application. By incorporating the material of the first aspect of the present invention into a matrix to form a filter material, the available active surface area is increased so as to aid efficiency. Furthermore, dispersion of the material in the contaminated fluid is reduced because it is held within the matrix. In some embodiments, webs of the filter matrix are loaded to a density of around 1kgm^{-2} ; a density of $.925\text{kgm}^{-2}$ has been found to be particularly effective in certain circumstances. In other embodiments, a density of around 0.5kgm^{-2} has been found to be effective, particularly where the web of filter matrix has a thickness in the region of 3 or 4mm . Once

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the filter material has become saturated with pollutants, it is relatively simple to remove the filter and replace it with a new one.

The filter material can carry over twice its own weight in hydrocarbons and is therefore useful as an energy source in, for example, cement kilns. In some situations, used filter material may be macerated into a form which can be fluidised and injected into furnaces as a fuelstock.

The filter material may be cut into shapes, such as discs, suitable for installation in filtration apparatus. Particularly preferred is a filter column comprising a hollow core upon which is mounted an alternating stack of filter plates and discs of the filter material of the third aspect of the present invention, wherein the filter plates are adapted to allow passage of fluid from a circumferential region of the filter column to the hollow core by way of the discs of filter material.

Alternatively, filter cartridges may be constructed by wrapping layers of the filter material around a central core. For example, a cylindrical cartridge may be made by taking a perforated tubular core, wrapping this with layers of filter material, providing a protective outer layer which allows passage of fluid into or out of the cartridge, and securing the whole with a pair of end caps. Fluid may be passed through the filter material in the cartridge either from the central core to the outside or from the outside to the central core.

One or more such filter cartridges may be advantageously installed in a filter pod comprising a casing internally divided into two chambers by a carrier which supports at least one filter cartridge, the carrier and the at least one cartridge being arranged so that fluid can only pass from one chamber

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to the other by passing through both the hollow tubular core and the filter material of the at least one cartridge.

It is also possible to fill the aforementioned filter cartridges directly with the material of the first aspect of the present invention, without employing a filter matrix.

According to a fourth aspect of the present invention, there is provided a method of cleaning a fluid by contacting the fluid with a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms, wherein one or more cellulosic materials are mixed together with a powdered formulation of the one or more carboxylic acids.

Fluids which may be cleaned by this method include gases such as air, and liquids such as water. The material may be in loose form, such as a material according to the first aspect of the present invention, or the material may be bound in a matrix according to the third aspect of the present invention.

For a better understanding of the present invention and to show how it may be carried into effect, reference shall now be made, by way of example, to the accompanying drawings, in which:

FIGURE 1 shows a section through a yard-scale filter rig incorporating the material of the first aspect of the present invention;

FIGURE 2 is an exploded view of a filter column incorporating the filter material of the third aspect of the present invention;

FIGURE 3 shows a detail of a filter plate from Figure 2;

FIGURE 4 shows a test circuit including a filter

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column similar to that of Figure 2;

FIGURE 5 shows a filter cartridge incorporating the filter material of the third aspect of the present invention;

FIGURE 6 shows a pod incorporating an array of the filter cartridges of Figure 5;

FIGURE 7 is a section through the pod of Figure 6;

FIGURE 8 shows a filter skid incorporating two of the pods of Figures 6 and 7;

FIGURE 9 shows a filter skid incorporating eight of the pods of Figures 6 and 7; and

FIGURE 10 is a graph showing the trends of flow/contamination data plotted against intervention periods for an eight-pod filter skid.

Initial small-scale trials:

There will firstly be described the results of small-scale laboratory analysis of a material according to the first aspect of the present invention. The differential pressure across columns of the material made from different types of cellulose was measured to gauge permeability and porosity characteristics of various substrates. The chosen grade showed no increase in differential pressure after 40 days flow. A particularly suitable grade of wood chip was found to be chips of a soft wood with a 30mm mean particle size.

Laboratory scale hydrocarbon filtration tests confirmed the oil-removing properties of the material. Two examples of the tests used follow:

Example 1: Dissolved hydrocarbons removed via column packed with 12g of material

575ml of Inde K condensate was added to 10 litres of artificial formation water (Brent recipe) and vigorously stirred using a homogeniser. Separate litre samples were then drained off, 100ml of each being

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retained for triple peak analysis to measure the dissolved hydrocarbon content before filtration, and the remainder being added to a litre separation funnel mounted above the column. Triple peak analysis was carried out on the filtered water to provide before and after figures. Results are shown in table 1:

Table 1

Sample Litre	% Dissolved Hydrocarbons Removed
1st	86
2nd	72
3rd	83
4th	67
5th	82
6th	59
7th	75
8th	75

Example 2: Treatment of dry salt contaminated with hydrocarbons from a desalination plant

Batches of salt were rehydrated by adding 0.3kg of salt to 1 litre of pure water and passed through 12g of material at the rate of 1 litre/minute. The results are shown in tables 2 and 3.

Table 2

Hydrocarbons/ppm		
	Before	After
Sample 1	2826	2
Sample 2	4481	3

Table 3

Hydrocarbons/ppm				
	Before		After	
	Disp.	Diss.	Disp.	Diss.
Sample 3	1110	18	1	0
Sample 4	636	13	1	0

This small-scale analysis served to identify the physical nature of the substrate that retained permeability over long periods, absorbed high levels of hydrocarbons and was 100% incinerable. Accordingly, larger, yard scale trials could now commence:

Filtering trials through a yard scale filter:

Figure 1 shows a yard scale rig that is used in the following analysis. The rig comprises a pump (1) which pumps contaminated water via a flowmeter (2) to a diffuser (3) mounted over a filtration bed (4) provided with an amount of the material (5) according to the first aspect of the present invention. The filter bed (4) is located within a receptacle (6), from which the filtered water is passed via a sample valve (7) to a discharge (8).

Example 3:

Real produced water from Sean Papa platform was supplied and batches of 1000 litres were filtered through 0.5kg of material at 10 litres/minute. Samples were taken before each batch and samples of filtered water taken every 10 minutes. Typical results are set out in tables 4 and 5.

Table 4

Sample	Hydrocarbons/ppm		
	Dissolved	Total	% Reduction
Pre filter B	1870	26723	
Filtrate B1	20	338	98
Filtrate B2	15	453	98
Filtrate B3	11	432	99
Filtrate B4	10	381	99
Filtrate B5	7	367	99
Filtrate B6	8	374	99
Filtrate B7	6	354	99
Filtrate B8	2	341	99
Filtrate B9	6	432	98
Filtrate B10	1	314	99

Table 5

Sample	Hydrocarbons/ppm		
	Dissolved	Total	% Reduction
Pre filter D	211	1409	
Filtrate D1	9	28	98
Filtrate D2	5	27	98
Filtrate D3	4	25	99
Filtrate D4	3	28	99
Filtrate D5	4	27	99
Filtrate D6	4	28	99
Filtrate D7	2	27	99
Filtrate D8	2	28	99
Filtrate D9	2	27	98
Filtrate D10	1	26	99

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In both these trials, the water had not been passed through any prefilter, coalescers or centrifuges, and therefore the total hydrocarbon levels (26723 - 1409ppm) were higher than expected. However, in a single pass through the filter, total hydrocarbon levels were reduced by an average >90%.

Specifically the dissolved fraction was reduced by an average 92%.

Typical suspended solids were 245ppm.

Formation of filter sheets:

There will now be discussed the use of the filter material of the third aspect of the present invention.

A granular formulation of a material according to a first aspect of the present invention is passed through a system that binds the loose product to, for example, a viscose or cotton gauze producing a 3mm thick mat that can be cut into shapes (typically discs) to fit existing filter hardware.

The process of forming such sheets increases the available surface area of the material thereby improving performance.

A prolonged programme of trials was undertaken to measure the performance of production grade discs in a commercially viable filtering system. The discs were installed in a filter column of the type shown in Figure 2, which comprises an alternating stack of filter plates (9) and discs (10) of filter material mounted on a hollow central core (12), the whole being compressed by an end plate (11) and placed in a suitable container (not shown). Figure 3 shows a filter plate (9) in more detail. Contaminated fluid is applied to the filter column such that it passes into the outside rim of each filter plate (9) and thence through an adjacent filter disc (10) before passing into the hollow central core (12) for extraction.

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Example 4:

2000 litres of synthetic produced water with 22ppm suspended solids (median size 2.5 - 19 μ m) was filtered through a disc filter column of the type shown in Figure 2 loaded with 16 discs (10) of filter material, samples being taken before and after filtering. Two tests were run using two different flow rates, series I at 48 litres/minute and series II at 10 litres/minute, the results being presented in tables 6 and 7, with table 8 showing the analysis of the condensate supplied.

Table 6

Series I Hydrocarbon removal

48 litres/minute

ppm					
		Dissolved	Dispersed	Total	Reduct -ion
					%
	Pre filter	27	489	516	
Minutes	10	6	10	16	97
Minutes	15	5	17	22	96
Minutes	20	8	19	27	95
Minutes	25	8	34	42	92

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Table 7

Series II Hydrocarbon removal

10 litres/minute

ppm					
		Dissolved	Dispersed	Total	Reduct -ion
					%
	Pre filter	52	792	844	
Minutes	10	6	11	17	98
Minutes	15	13	15	28	97
Minutes	20	6	16	22	97
Minutes	25	6	15	21	98

Table 8:

Condensate Composition

Component	%age Weight
C1	<0.01
C2	0.04
C3	0.16
i-C4	0.17
n-C4	0.33
neo-C5	0.04
i-C5	0.37
n-C5	0.45
cyclo-C6	0.08
C6	1.99
Benzene	1.62

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C7	3.55
Toluene	2.08
C8	3.70
Ethyl-benzene	0.53
Xylenes	3.42
C9	3.75
Isopropylbenzene	0.98
1,2,4 Trimethylbenzene	1.90
C10	6.65
C11	9.74
C12	8.90
C13	9.52
C14	8.96
C15	8.10
C16	5.23
C17	4.80
C18	3.54
C19	2.48
C20	1.78
C21	1.32
C22	0.98
C23	0.69
C24	0.60
C25	0.37
C26	0.34

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C27	0.32
C28	0.26
C29	0.19
C30	0.07

5

In the following examples, the object was to establish the time taken for a given quantity of filter material discs to become saturated with hydrocarbons and to monitor continuously the hydrocarbon removal performance. These trials used fluorescing on-line hydrocarbon monitoring instruments.

10

Example 5:

A 16 disc filter using a prototype grade of material according to the first aspect of the present invention was used to treat 500ppm condensate in water. This was made up by homogenising the oil into a static water volume of 25 litres.

15

A laboratory fluorometer recorded:

20

Run 1 93.5% removal at 5 litres/minute
Run 2 96% removal at 5 litres/minute
Run 3 >87% removal at 5 litres/minute

Changes were made to the means of dispersing the oil in water as shown in the circuit of Figure 4, which comprises a contaminant dosing pump (13), a seven-stage homogenising pump (14), a filter column (15) and a sampling reservoir (16), wherein the sampling reservoir (16) generates an overflow stream and a monitored stream, the latter stream passing through an on-line monitor (17) adapted to detect the presence of hydrocarbons by fluorescence.

25

30

Production grade filter discs were manufactured for future tests to a repeatable commercial production formulation with a weight of 1000gm⁻².

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Example 6:

In this example, the filter column and discs used in Example 5 were reused. The 16 disc filter was connected on-line with a process monitor calibrated to the exact condensate type. The hydrocarbon-polluted stream was pumped through the 16 disc filter after dynamic mixing.

Contaminants were injected into the influent water at a constant rate through a positive displacement pump directly into the throat of the homogenising pump. The outlet pressure was controlled to 98psi. Samples for on-line analysis were taken directly through the falling stream cell; samples for laboratory analyses were taken before and after filtration.

For the first continuous test the metering of condensate was calibrated to 500ppm in water with a mean droplet size of $<10\mu\text{m}$. Over the next two hours the filter continued to remove $>90\%$ of the condensate in water supplied at a rate of 8 litres/minute. The test circuit shown in Figure 4 proved to be a satisfactory system for future trials to test efficiency and filter capacity.

Production grade filter discs were used in the following tests:

Example 7:

A nominal 300ppm condensate in water was passed at 4 litres/minute through a fresh filter column, the results being presented in table 9.

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Table 9

Elapsed Time (h)	Through Filter (l/min)	Monitored Inlet Conc./ppm	Measured Outlet Conc./ppm	% Removal
0.00	4.02	312	16	95
2.50	4.05	305	18	94
3.75	4.05	310	20	94

Example 8:

This was a repeat of the 300ppm test at 4 litres/minute but using new filter discs. This test ran for seven hours before showing signs of reaching saturation, the results being presented in table 10.

Table 10

Elapsed Time (h)	Through Filter (l/min)	Monitored Inlet Conc./ppm	Measured Outlet Conc./ppm	% Removal
0.00	4.02	310		
1.00	4.02	307	6	98
1.50	4.00	295	6	98
2.25	3.97	312	8	97
3.25	4.00	312	10	97
4.25	4.00	308	12	96
5.25	4.05	310	13	96
7.00	3.98	312	18	94

Example 9:

A nominal 500ppm condensate was passed through the above filter stack at the same volumetric flow rate so as to accelerate saturation, the results being presented in table 11. The filter column is showing signs of failure.

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Table 11

Elapsed Time (h)	Through Filter (l/min)	Monitored Inlet Conc./ppm	Measured Outlet Conc./ppm	% Removal
7.00	4.05	498	18	96
7.25	4.05	498	18	96
8.00	4.03	496	35	93
8.25	4.03	498	88	82

Example 10:

A nominal 500ppm condensate at 6 litres/minute was passed through the filter stack, the results being presented in table 12.

Table 12

Elapsed Time (h)	Through Filter (l/min)	Monitored Inlet Conc./ppm	Measured Outlet Conc./ppm	% Removal
0.00	6.51	515		
0.50	6.32	515	11	98
1.00	6.42	515	14	97
2.00	6.35	513	14	97
3.00	6.42	516	22	96
3.50	6.46	514	26	95
4.50	6.42	514	46	91
5.67	6.42	513	62	87

Example 11: Glycol removal

A larger filter housing using 18" filter discs was connected to the test rig shown in Figure 2. Glycol contaminated with hydrocarbons was supplied, this mixture was injected to the total stream of 14 litres/minute.

This level of contamination is beyond the

-18-

calibration range of the standard on-line monitor, but it was possible to use the 4-20mA output to monitor changes in the inlet flow to the filter.

Hand samples were taken for analysis before and after filtering. The results are shown in table 13.

Throughout the test the filters were operating at 17psi. The filter elements were on-line for 150 minutes.

Table 13

	ppm		%
	Prefilter	Post filter	Reduction
Glycol MEG	4520	797	82
Glycol MEG	4566	831	82
Dispersed hydrocarbons	95	0	100
Dissolved hydrocarbons	7	0	100

Example 12: Metals removal

In this example, water with the following levels of metals was used:

Nickel 1.75ppm

Copper 0.50ppm

Zinc 0.40ppm

Lead 0.30ppm

and the following was added to the water:

Condensate 0.5ppm

Leman silt 50ppm

Samples were taken before and after filtering, the results being shown in table 14. The increase in the metal content of the prefiltered water is ascribed to metals in the condensate and silt.

Table 14

	ppm		%
	Prefilter	Post filter	Reduction
Nickel	1.77	0.52	71
Copper	0.62	0.14	71
Zinc	2.9	0.25	57
Lead	0.42	<0.05	88

Formation of filter cartridges:

As an alternative to the disc filter column described above, a pod including one or more filter cartridges may be employed. As shown in Figure 5, a cartridge (18) is formed by wrapping filter material (19) around a tubular core (20) and applying a protective outer layer (21) and end caps (22). The protective outer layer (21) and the core (20) are provided with perforations (23,24) so as to allow passage of filtrate, either from the outside of the cartridge (18) through the filter material (19) to the inside of the core (20) or vice versa.

A typical cartridge (18) has a diameter of 215mm, a length of 1300mm and an internal diameter of 50mm, and is provided with 10.98kg of tightly-wrapped sheet filter material with a density of 0.925kgm⁻².

Example 13:

Figures 6 and 7 show a pod (25) in which an array of filter cartridges (18) is mounted on a carrier (26). The carrier (26) is adapted to allow filtrate ingressing from the bottom (27) of the pod (25) to pass only into the hollow cores (20) of the cartridges (18). The filtrate then passes through the filter material (19) and into the top (28) of the pod (25) (which is separated from the bottom (27) of the pod (25) by the carrier (26)), from where it exits the pod (25) by way of egress (29).

Example 14:

Figure 8 shows a filter skid (30) comprising two filter pods (25) and Figure 9 shows a filter skid (31) comprising eight filter pods (25). Figures 8 and 9 are not to scale. The pods (25) may be connected in series or in parallel in a number of configurations by way of alternative manifolding of the connecting pipework (32). A typical eight-pod skid (31) can accommodate flow rates of up to 120m³/hour at working pressures of up to 4 bar.

Table 15 shows the results achieved for an eight-pod skid (31), each pod (25) comprising ten filter cartridges (18) and operated according to the following specifications:

Weight of filter material per cartridge: 10.98kg
 Weight of filter material per vessel: 109.8kg
 Weight of filter material in skip: 878.4kg
 Oil contamination: 500mg/litre
 Oil adsorption: 90%
 MEG contamination: 500mg/litre
 MEG adsorption: 80%
 Flow: 1656 litres/hour

Table 15:

Flow litres/hour	Discharge			Retained contaminants		Interv- ention
Through filter	mg/l	kg/day	kg/annum	mg/l	kg/day	Days
1656	150	5.96	2176	850	33.79	52.00

Figure 10 shows a plot of the trends of flow rate against contamination and intervention periods for a typical eight-pod filter skid employing the filter material of the third aspect of the present invention.

CLAIMS:

1. A filter material comprising a matrix in which is dispersed a granular formulation of a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms.

2. A material as claimed in claim 1, wherein the one or more aliphatic carboxylic acids have hydrocarbon chains consisting of 10 to 18 carbon atoms.

3. A material as claimed in claim 1 or 2, wherein the one or more carboxylic acids are selected from the group comprising stearic acid and palmitic acid.

4. A filter material as claimed in claim 1, 2 or 3, wherein the matrix comprises a non-woven fibrous material.

5. A filter material as claimed in claim 1, 2 or 3, wherein the matrix comprises an open-cell foam materials.

6. A filter material as claimed in claim 1, 2 or 3, wherein the matrix comprises a cotton or viscose gauze.

7. A filter column comprising a hollow core upon which is mounted an alternating stack of filter plates and discs of the filter material as claimed in any of claims 1 to 6, wherein the filter plates are adapted to allow passage of fluid from a circumferential region of the filter column to the hollow core by way of the discs of filter material.

8. A filter cartridge comprising a hollow core around which is wrapped one or more layers of a filter material as claimed in any one of claims 1 to 6.

9. A filter pod comprising a casing internally divided into two chambers by a carrier which supports at least one filter cartridge as claimed in claim 8,

the carrier and the at least one cartridge being arranged so that fluid can only pass from one chamber to the other by passing through both the hollow tubular core and the filter material of the at least one cartridge.

10. A method of producing a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms, wherein one or more cellulosic materials are mixed together with a powdered formulation of the one or more carboxylic acids.

11. A method according to claim 12, wherein the one or more cellulosic materials are selected from the group comprising wood chips and virgin pulp.

12. A method according to claim 10 or 11, wherein latex is added to the one or more cellulosic materials and the one or more carboxylic acids.

13. A method according to any one of claims 10 to 12, wherein mixing takes place in a hammer mill.

14. A method of cleaning a fluid by contacting the fluid with a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms, wherein one or more cellulosic materials are mixed together with a powdered formulation of the one or more carboxylic acids.

15. A method according to claim 14, wherein the fluid is air.

16. A method according to claim 15, wherein the fluid is water.

17. A filter cartridge comprising a container having a fluid input and a fluid output and including therebetween a quantity of the material of any one of claims 1 to 6.

ABSTRACT

There is disclosed a material comprising a base formed substantially of cellulose fibers onto which is absorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms, and a filter material comprising a matrix in which is dispersed a granular formulation of this material. The material can be used to adsorb hydrocarbon pollutants, such as oil spills, and the filter material can be used to adsorb such pollutants from a fluid stream.

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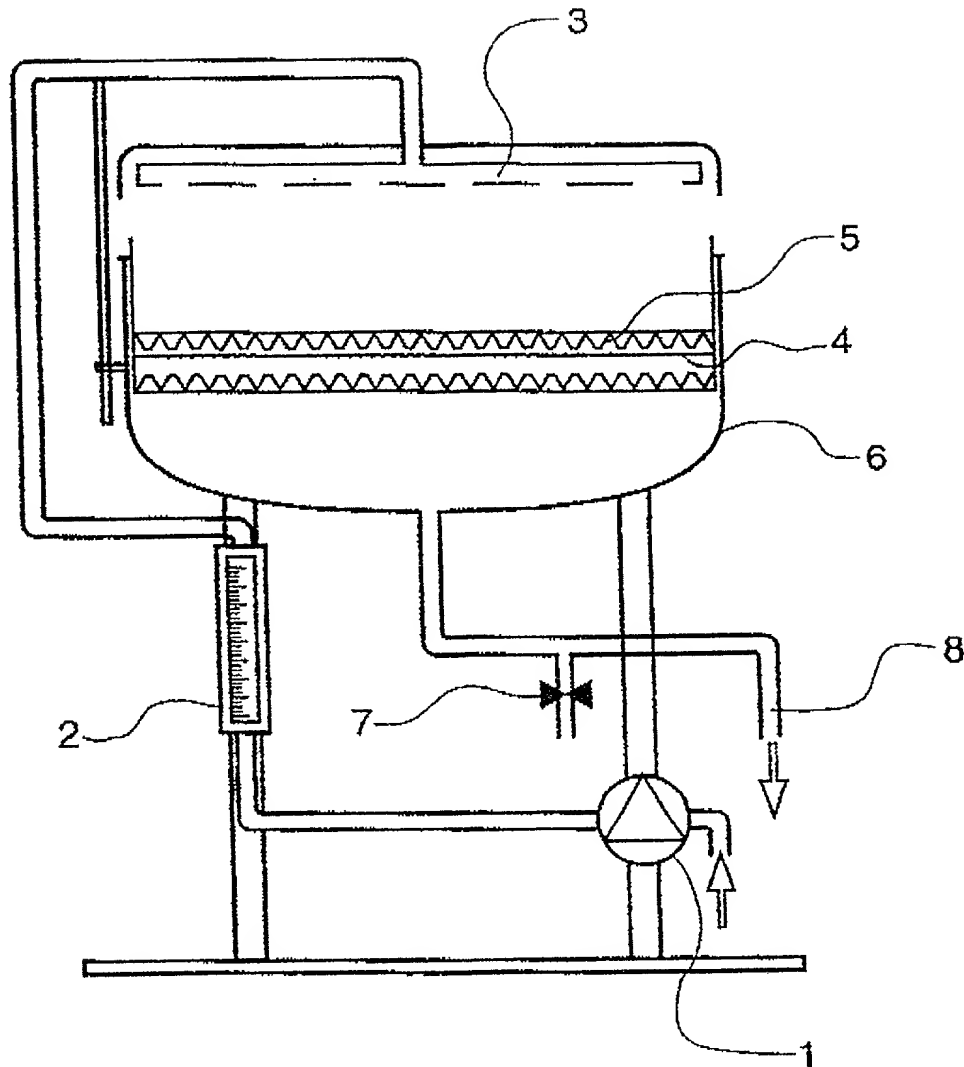


Fig. 1

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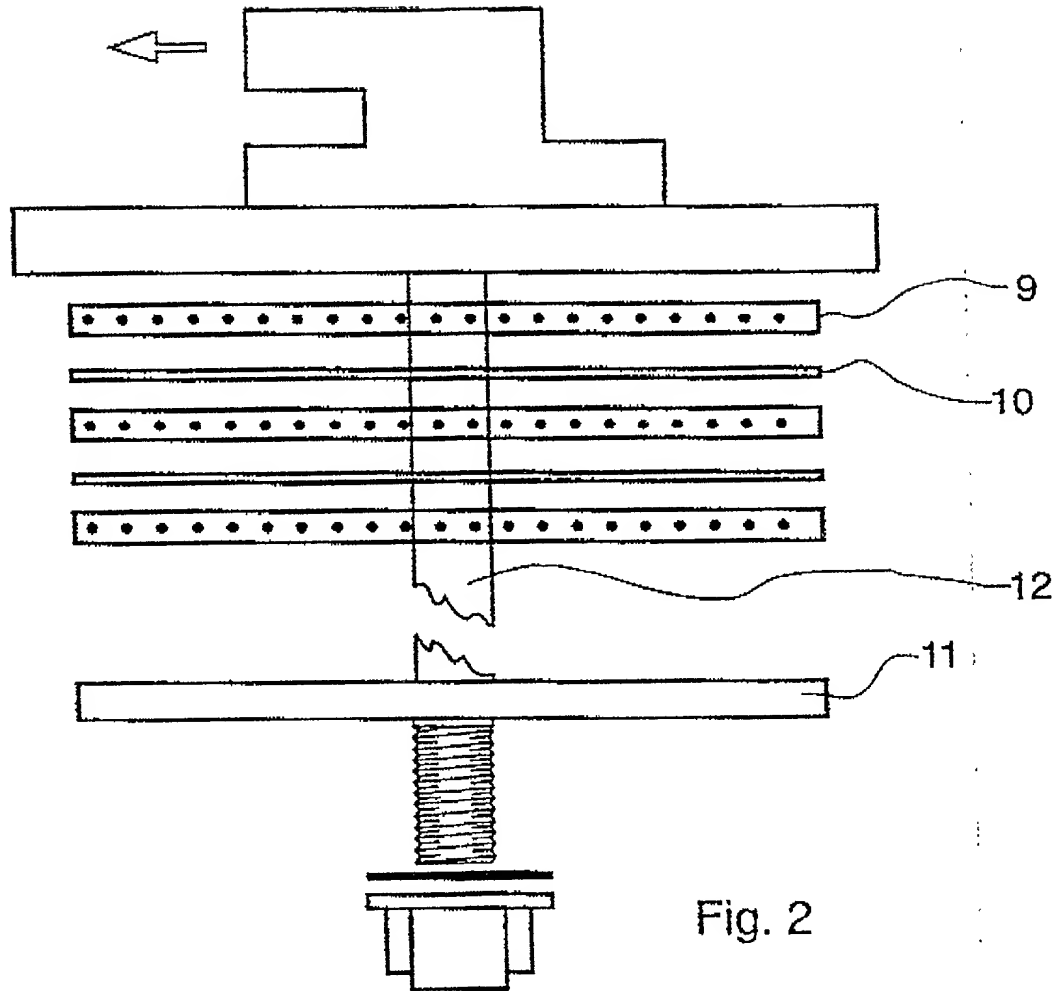


Fig. 2

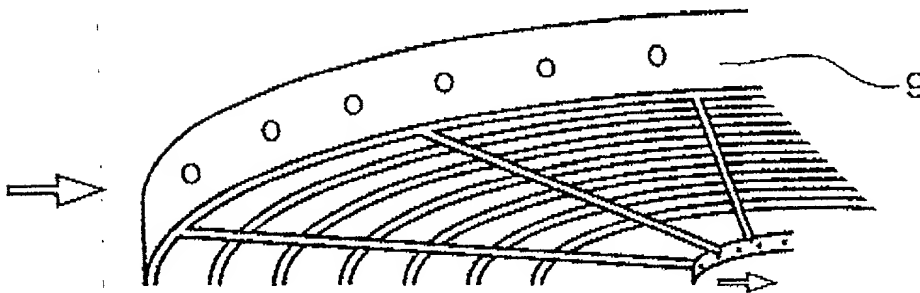


Fig. 3

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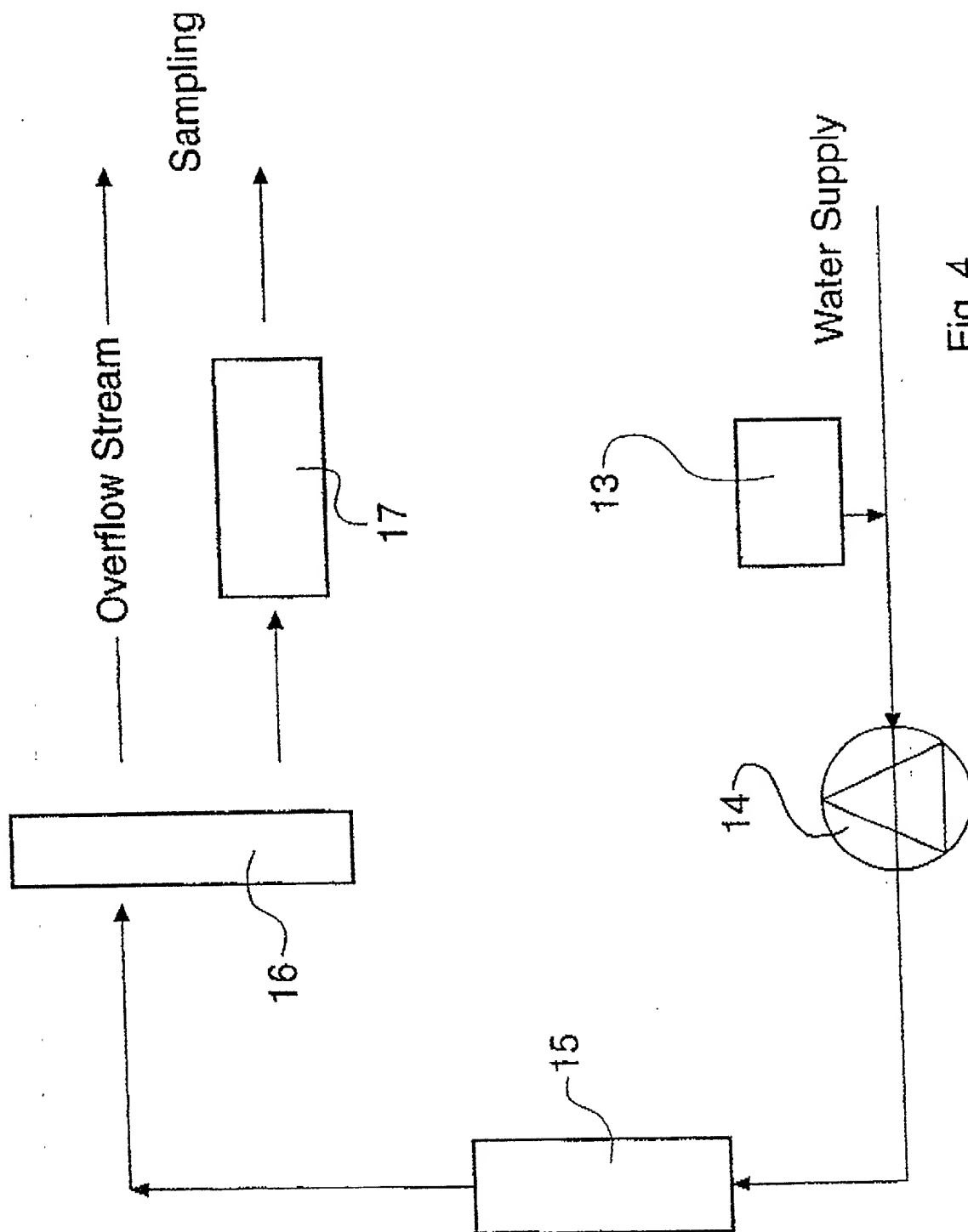


Fig. 4

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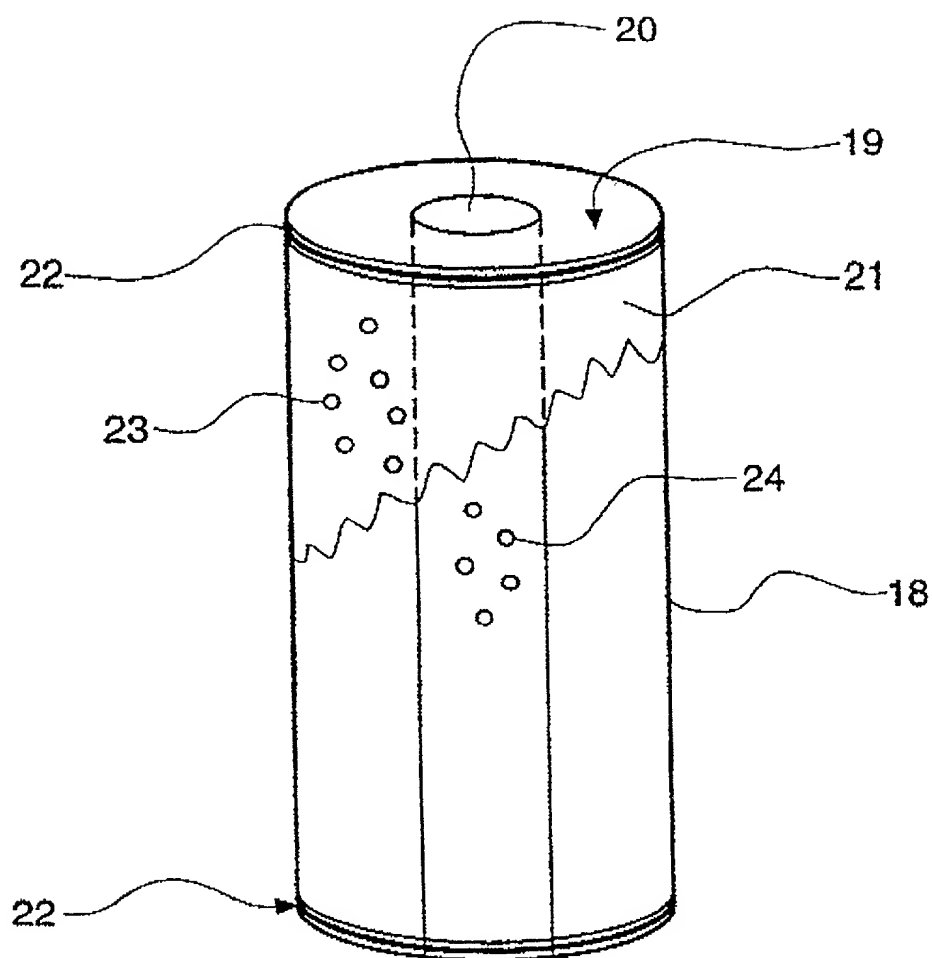


Fig. 5

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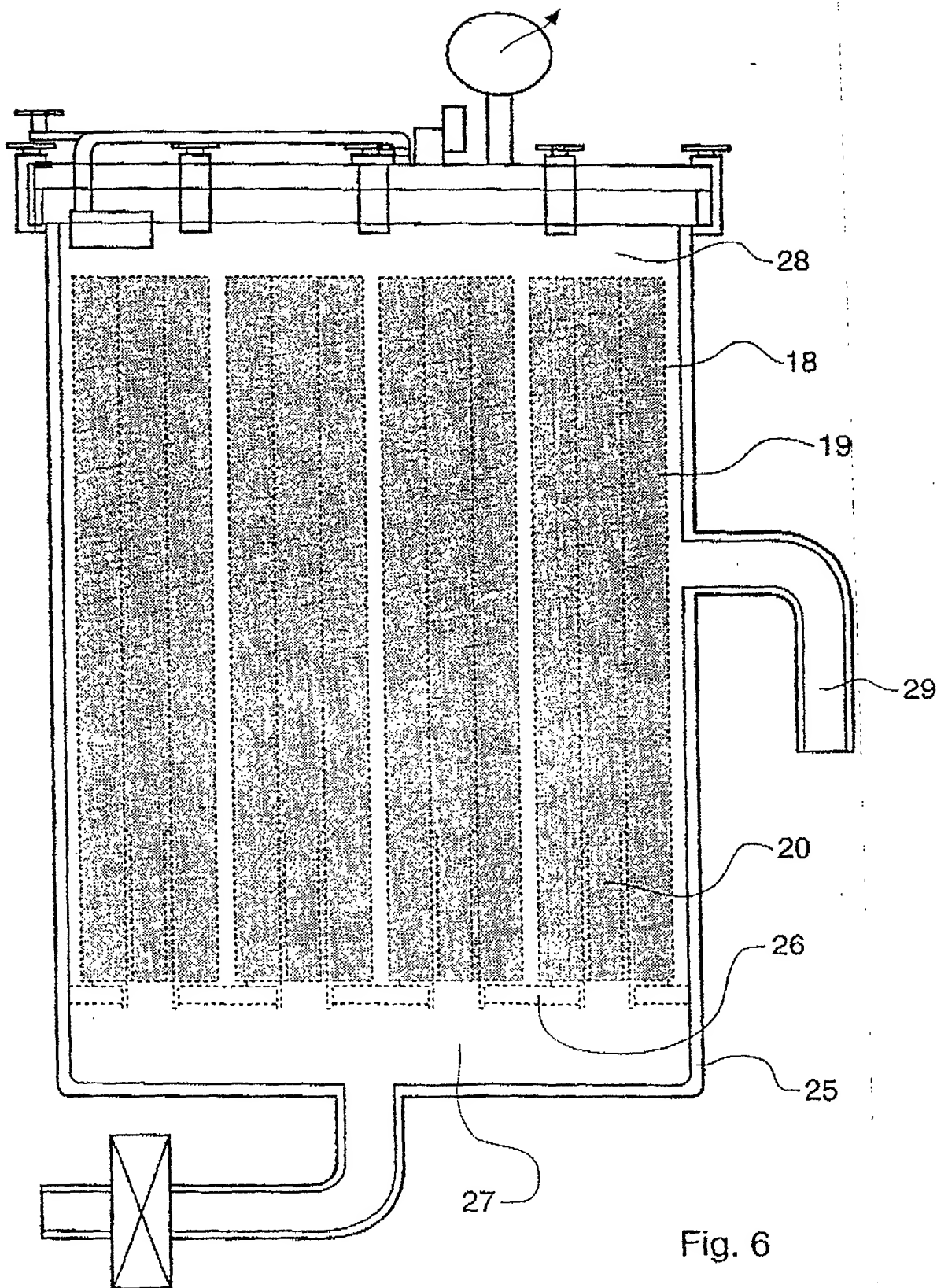


Fig. 6

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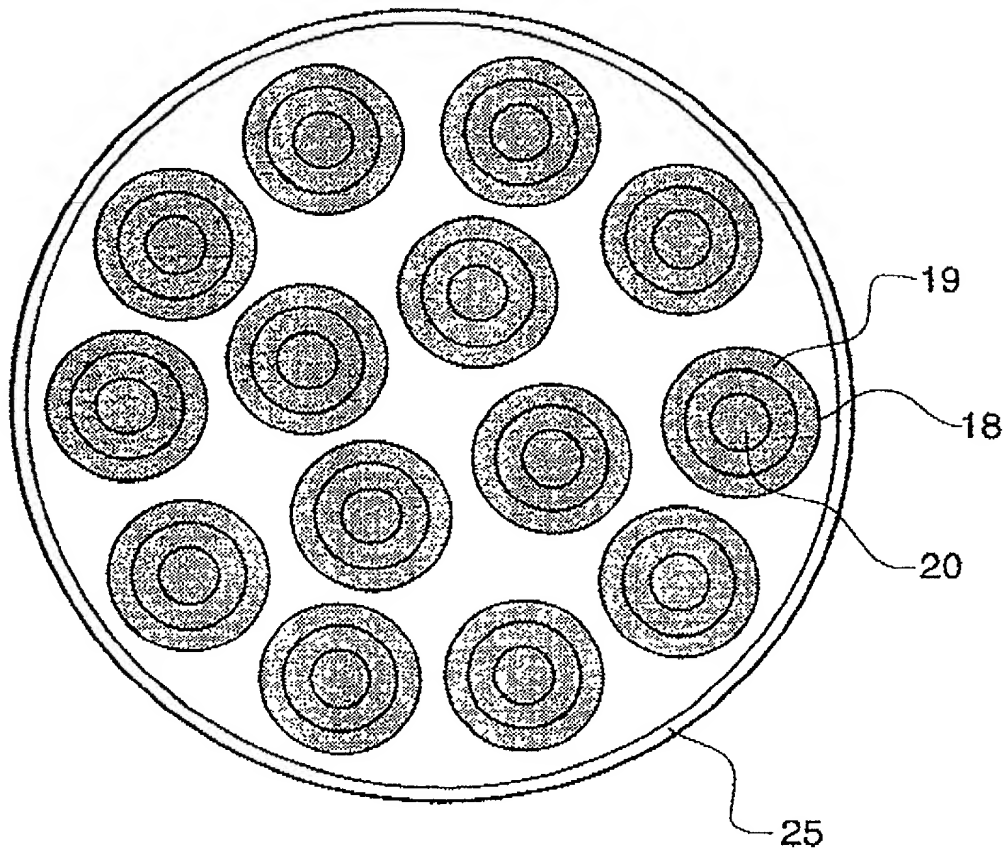


Fig. 7

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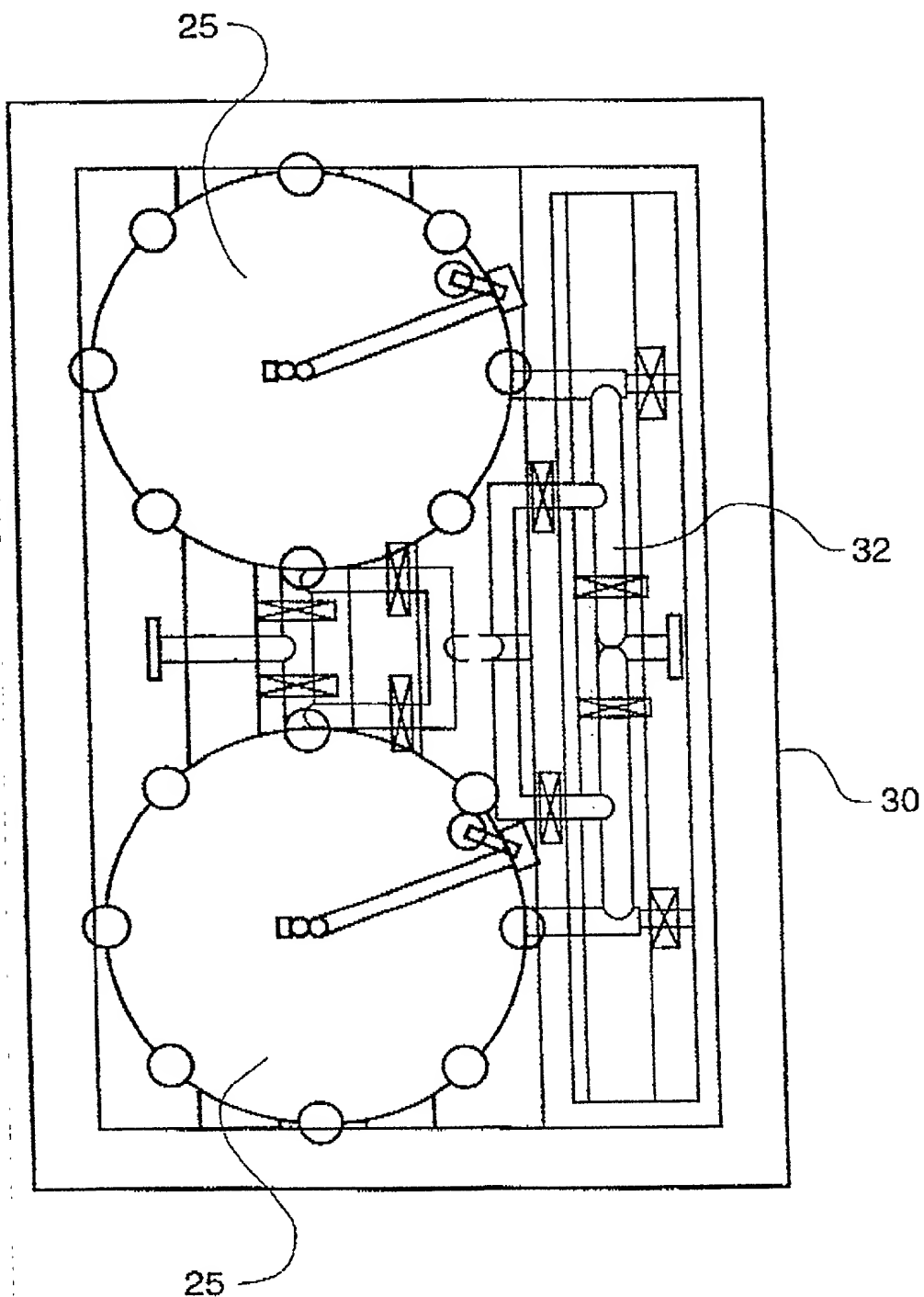


Fig. 8

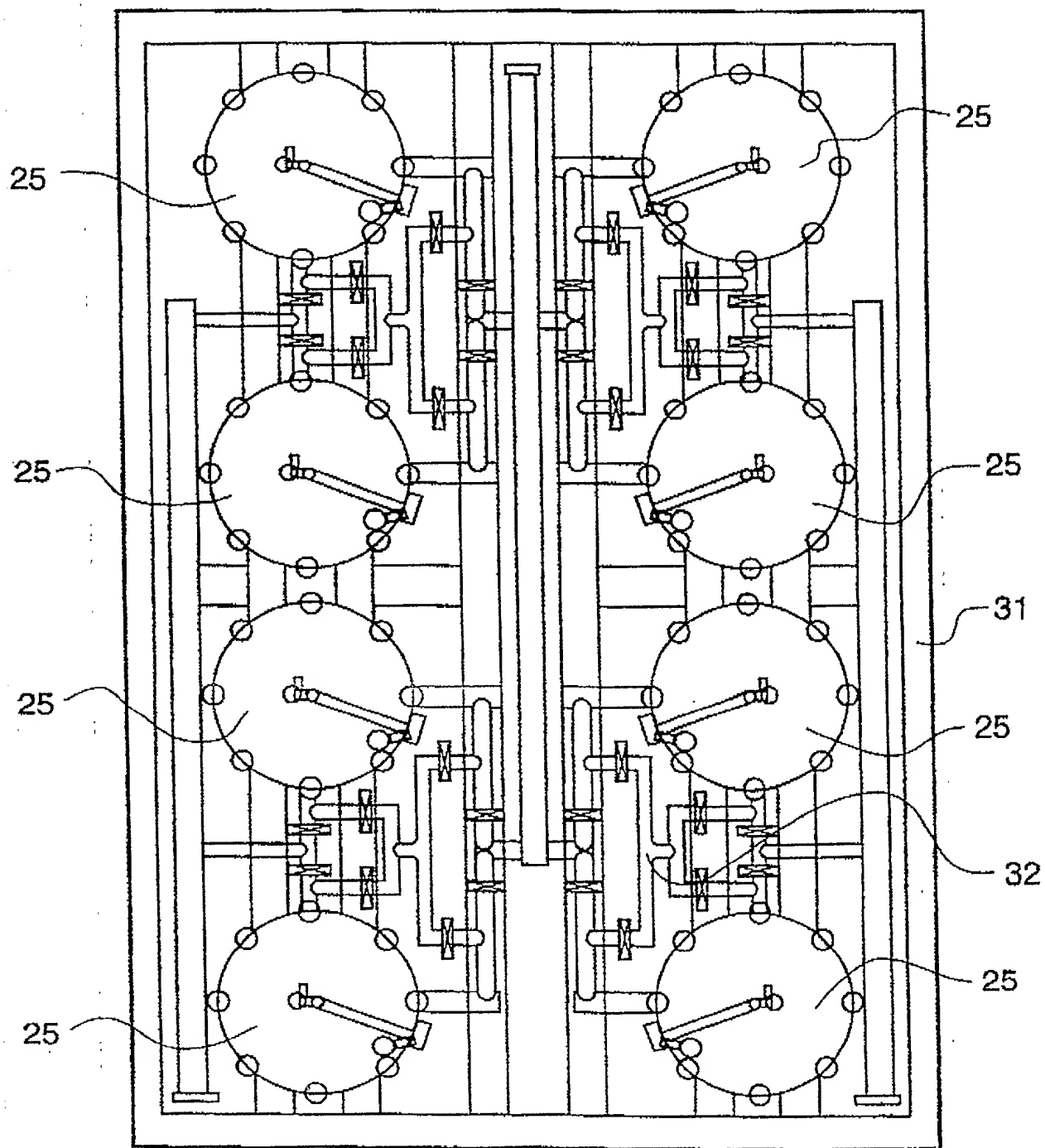
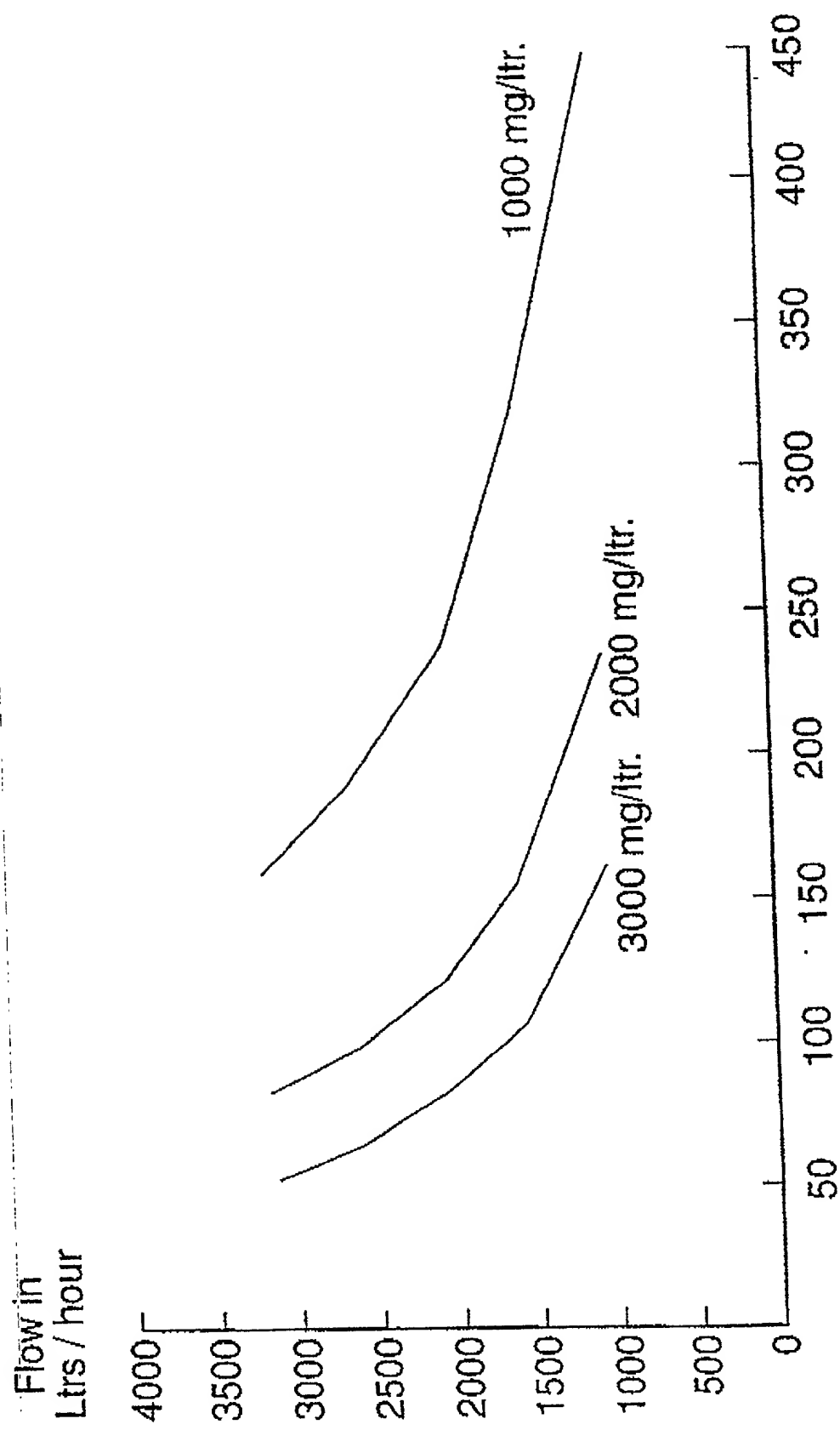


Fig. 9

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Time Between Intervention in Hours Fig. 10

Combined Declaration for Patent Application and Power of Attorney

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name; and that I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

LIQUID AND GAS PURIFICATION AND FILTRATION

the specification of which (check one)

[] is attached hereto;

[] was filed in the United States under 35 U.S.C. §111 on _____, as
USSN _____*; or

[XX] was/will be filed in the U.S. under 35 U.S.C. §371 by entry into the U.S. national stage of an international (PCT) application, PCT/GB97/01319, filed MAY 14, 1997, entry requested on December 16, 1998*; national stage application received

USSN 09/202,500 *; §371/§102(e) date _____* (*if known),

and was amended on December 16, 1998 (if applicable).

(include dates of amendments under PCT Art. 19 and 34 if PCT)

I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above; and I acknowledge the duty to disclose to the Patent and Trademark Office (PTO) all information known by me to be material to patentability as defined in 37 C.F.R. § 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. §§ 119, 365 of any prior foreign application(s) for patent or inventor's certificate, or prior PCT application(s) designating a country other than the U.S., listed below with the "Yes" box checked and have also identified below any such application having a filing date before that of the application on which priority is claimed:

<u>9610215.7</u>	<u>Great Britain</u>	<u>16 May 1996</u>	[X]	[]
(Number)	(Country)	(Day Month Year Filed)	YES	NO
_____	_____	_____	[]	[]
(Number)	(Country)	(Day Month Year Filed)	YES	NO

I hereby claim the benefit under 35 U.S.C. § 120 of any prior U.S. non-provisional Application(s) or prior PCT Application(s) designating the U.S. listed below, or under § 119(e) of any prior U.S. provisional applications listed below, and, insofar as the subject matter of each of the claims of this application is not disclosed in such U.S. or PCT application in the manner provided by the first paragraph of 35 U.S.C. §112, I acknowledge the duty to disclose to the PTO all information as defined in 37 C.F.R. §1.56(a) which occurred between the filing date of the prior application and the national filing date of this application:

_____ (Application Serial No.)	_____ (Day Month Year Filed)	_____ (Status: patented, pending, abandoned)
_____ (Application Serial No.)	_____ (Day Month Year Filed)	_____ (Status: patented, pending, abandoned)
_____ (Application Serial No.)	_____ (Day Month Year Filed)	_____ (Status: patented, pending, abandoned)

I hereby appoint the following attorneys, with full power of substitution, association, and revocation, to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith.

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The undersigned hereby authorizes the U.S. Attorneys or Agents named herein to accept and follow instructions from Haseltine Lake as to any action to be taken in the U.S. Patent and Trademark Office regarding this application without direct communication between the U.S. Attorney or Agent and the undersigned. In the event of a change of the persons from whom instructions may be taken, the U.S. Attorneys or Agents named herein will be so notified by the undersigned.

U.S. Application filed _____, Serial No. 09/202,500

PCT Application filed MAY 14, 1997, Serial No. PCT/GB/97/01319

I hereby further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

13 MAR 1999

1-20 FULL NAME OF FIRST INVENTOR Andrew Bickford HAYNS		INVENTOR'S SIGNATURE <i>X Andrew Hayns</i>		DATE <i>X 12/3/99</i>
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RESIDENCE			CITIZENSHIP	
POST OFFICE ADDRESS				
FULL NAME OF FIFTH JOINT INVENTOR		INVENTOR'S SIGNATURE		DATE
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POST OFFICE ADDRESS				
FULL NAME OF SIXTH JOINT INVENTOR		INVENTOR'S SIGNATURE		DATE
RESIDENCE			CITIZENSHIP	
POST OFFICE ADDRESS				
FULL NAME OF SEVENTH JOINT INVENTOR		INVENTOR'S SIGNATURE		DATE
RESIDENCE			CITIZENSHIP	
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ALL INVENTORS MUST REVIEW APPLICATION AND DECLARATION BEFORE SIGNING. ALL ALTERATIONS MUST BE INITIALED AND DATED BY ALL INVENTORS PRIOR TO EXECUTION. NO ALTERATIONS CAN BE MADE AFTER THE DECLARATION IS SIGNED. ALL PAGES OF DECLARATION MUST BE SEEN BY ALL INVENTORS.